CONSIDERATIONS IN THE DESIGN AND UTILIZATION OF SPUTTERING SYSTEMS

by

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TABLE OF CONTENTS

Chapter | Page

I. INTRODUCTION. 1

II. APPARATUS 7

III. AN ECONOMICAL SYSTEM. 15

IV. DEPOSITION RATES. 20

V. OTHER SPUTTERING PHENOMENA. 27

- Self-Bias Phenomenon 27
- High Pressure Phenomena. 29
- Contamination Phenomena. 31

VI. EXPERIMENTAL RESULTS. 34

- System Description 34
- Tuning the R. F. Discharge 35
- Substrate Stress 36
- High Pressure Effects. 38
- System Impedance Characteristics 40
- Conclusion 42

SELECTED BIBLIOGRAPHY. 43

ACKNOWLEDGMENTS. 45

LIST OF FIGURES

Figure Page

1. Block diagram of typical R. F. sputtering system. . . . 7
2. Original R. F. sputtering chamber arrangement 8
3. Typical R. F. sputtering chamber. 9
4. R. F. impedance matching network. 12
5. Equivalent circuit of an R. F. sputtering system. . . . 12
6. Sectional view of economical sputtering chamber 16
7. Completed sputtering chamber. 18
8. Lower base plate of sputtering chamber. 18
9. Variation in yield with angle of incidence. 21
10. Deposition rate vs. discharge power 23
11. Deposition rate vs. substrate temperature 24
12. Deposition rate vs. quality of ground 25
13. Deposition rate vs. magnetic field intensity. 26
14. Potential of target surface, V, vs. time. 27
15. Resistivity of Ta sputtered films versus sputtering voltage 30
16. Sputtering system 35
17. Substrate clamping arrangement. 37
18. Area of film non-adhesion due to substrate stress . . . 37
19. Impedance characteristics for D. C. system with Cu electrode 41

CHAPTER I

INTRODUCTION

As a method of thin film deposition, sputtering has recently enjoyed a rapid increase in utilization. It is not, however, a recently discovered process. Sputtering phenomena were first observed more than 100 years ago, in 1852, by Grove. The use of sputtering for film deposition is therefore merely an adaptation of a process which has been studied for over a century.

The physical process of sputtering may be defined as the ejection of atoms from a target surface under the influence of ion bombardment. Essentially, sputtering may be described as a process of atomic billiards which occurs primarily in the surface layers of the target. Inert or reactive gas atoms are ionized and accelerated to the target surface, where they eject particles of the target material.

Two main types of particles are ejected: heavy particles and secondary electrons. The heavy particles which are ejected may be neutral, positive ions or negative ions.

Mass spectrometric studies show that complex particle combinations are ejected from a surface under positive ion bombardment. Honig (9) observed 13 species of neutrals, 21 species of positive ions and 21 species of negative ions when he bombarded a contaminated SiC surface with 600 eV noble gas ions. (Only about one percent of the ejected heavy particles, however, were found to be ions.) Other studies of positive ion emission from copper, molybdenum, platinum and tantalum under the bombardment of inert gas ions found that between 0.1 and 1.0 percent of the ejected heavy particles were charged (12).

Studies involving compounds indicate that entire molecules may be sputtered. For instance, for the compound GaSb, it can be shown from energy considerations that removal of only the surface atom, whether Ga or Sb, results in an unstable configuration. Therefore, GaSb molecules must be sputtered. Data obtained by Wolsky (26) confirm this conclusion.

Similar studies have been made to determine ejected secondary electron yield (15). These studies reveal that γ_i, or the number of electrons per incident ion, ranges from about 0.01 at 100 eV ion energy to about 0.1 at 1 keV ion energy. (These results are valid for K, Li and Rb ions incident on Al, and for H and Na ions incident on Cu.) For 1-3 keV argon ions incident on W, Petrov and Hagstrum (10) have found that 0.1-0.2 secondary electrons are emitted per incident argon ion.

A process important to the understanding of the sputtering phenomenon is the process by which ionization is initiated in the sputtering chamber. Ionization can be initiated by what Francis (7) calls "secondary processes". These processes include emission of electrons from the cathode by the incidence of photons (γ_p).

Electrons may also be emitted thermally, since $T \gg 0^{o}K$ for the sputtering cathode, and in thermionic emission the electrons must achieve only enough energy to overcome the work function $e\phi$.

A third possibility is for electrons to be emitted from the surface by "field emission". Wave mechanical calculations show that an electron in a potential well, the target, has a finite probability of escaping through this barrier to a free state of the same potential energy. This probability increases as the applied field increases.

Photo-ionization, or photons impinging on the gas atoms, causing direct ionization, may also lead to breakdown. This process is more important at low values of X/p, where X = the field applied in volts per centimeter, and p = the chamber pressure in mm Hg.

Several collisions between electrons and gas atoms may be necessary to cause ionization if the electron energies are low, i.e. less than eV_i, the required energy for ionization. There thus may be many more exciting than ionizing collisions.

For R. F. sputtering, secondary processes at the electrodes are not absolutely necessary for breakdown to occur, as they are in D. C. discharges. This is because the field is reversing direction periodically, and the charges may not be swept out of the chamber onto the walls or the electrodes. With the resultant lower losses, much lower fields can lead to a growth of ionization, resulting in a self-sustained discharge. It should be noted, however, that for R. F. sputtering, the secondary electrons, whether produced by thermionic emission or photons, will not contribute to the growth of the discharge unless they are emitted when the field is in a favorable direction.

It follows from the above discussion that the total glow discharge current is $i_+ + \gamma_i i_+$, where i_+ is the positive ion current and γ_i is the number of electrons liberated from the target per positive ion.

Three main types of sputtering are presently used: diode sputtering, reactive sputtering and R. F. sputtering. Bias sputtering, which makes use of D. C. or R. F. to bias the substrate, has lately gained wide usage. It is, however, only a variation of the abovementioned main types.

Diode sputtering consists of establishing a direct current glow discharge between two parallel electrodes. These electrodes are a cathode which acts as the supply of material to be deposited, and an anode which contains the substrate to be coated. The sputtering is carried out in an inert gas atmosphere in a vacuum chamber. Typical parameters are: 1.5 - 7.0 kV voltage drop, 0.15 - 1.50 ma/cm^2 current density, 10 - 100 millitorr chamber pressure and 3 - 10 cm electrode spacing (17).

Reactive sputtering consists of adding a reactive gas (such as O_2, N_2, CO, CH_4, or H_2S) to the inert gas (usually argon) to form interstitial solutions of the reactive product in the deposited metal, or for the formation of individual compounds (8). The concentration of the reactive gas must be kept low in order to avoid the formation of insulating oxide films at the cathode.

R. F. sputtering consists of establishing a glow discharge by applying radio frequency energy to the cathode of a sputtering chamber. When sputtering metals, the R. F. is coupled to the cathode through a blocking capacitor. When sputtering insulators, the R. F. is coupled directly to the cathode. A chamber pressure of about five millitorrs is often used for R. F. sputtering.

The use of sputtering, regardless of type, frequently offers substantial advantages over other methods for the deposition of thin films. Sputtering allows controlled film formation over a very wide range of deposition rates, especially in the very low rate region where, for some materials, evaporation is difficult, if not impossible, to control. Films have been formed by sputtering at rates as low as one angstrom per minute (25).

The sputtering process yields very reproducable results. For instance, tantalum nitride resistors formed by reactive sputtering can be fabricated to initial tolerances as low as 0.01 percent (14).

Sputtered films exhibit very good mechanical properties. One reason is that the films are under compressive stress. Measurements have found stresses of 10^9 dynes/cm^2 for two micron thick SiO_2 films (4). Also, adhesion is better than is obtained by other deposition methods, and in general is excellent.

R. F. sputtering offers important advantages over D. C. and reactive sputtering. One of the most important advantages is that insulators can be sputtered, since, using R. F. energy, power can be fed into the system via the displacement current through the dielectric material. The insulator target is thus alternately bombarded with electrons and ions. Therefore, the positive charge which is accumulated during the negative (sputtering) part of each cycle is neutralized by electrons during the positive part of the cycle.

Several properties of R. F. sputtering were found to be advantageous for metals as well as for insulators. For example, the sputtering of refractory metals, which is difficult with D. C. methods, is easily done at high rates by R. F. sputtering. Attempts to deposit molybdenum films thicker than one micron by evaporation or D. C. sputtering are usually unsuccessful. However, molybdenum films three microns thick have been R. F. sputtered and subsequently processed without peeling (2).

A thorough understanding of the sputtering process has been limited to a great degree by the lack of reliable experimental data. One reason is that early experiments were carried out in relatively high pressure glow discharges. Subsequent chapters will examine recent experimental data and apparatus technology.

CHAPTER II

APPARATUS

The fabrication of sputtering systems is far from standardized. In fact, there are almost as many different equipment arrangements as there are systems. The block diagram of Figure 1, however, depicts a typical R. F. sputtering system. The arrangement for a D. C. system is essentially the same, with the substitution of a D. C. power supply for the R. F. generator and impedance matching network.

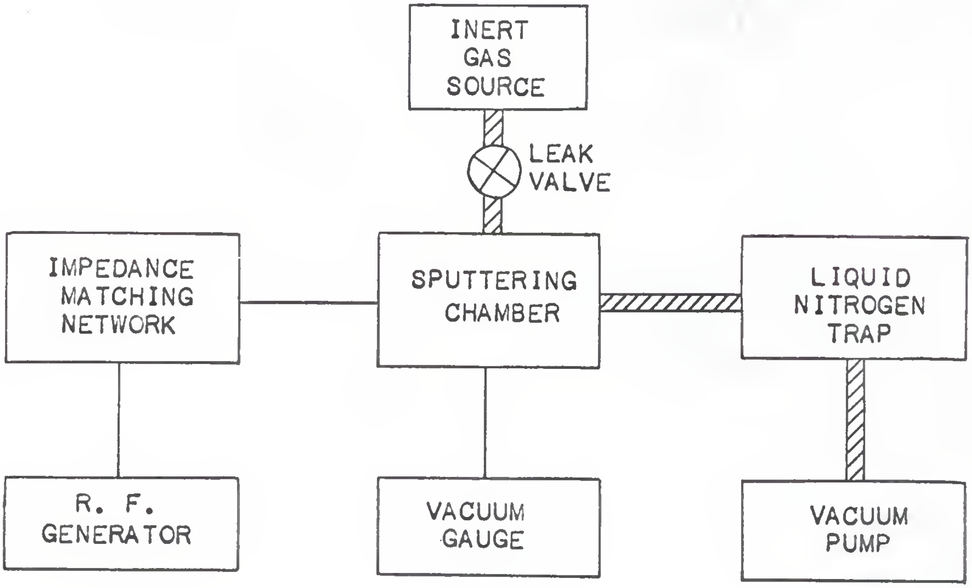

Figure 1. Block diagram of typical R. F. sputtering system.

When designing a sputtering system, consideration must be given to each element of Figure 1. Perhaps the most careful consideration must be given to the sputtering chamber itself.

Although system configurations have changed substantially since Anderson, Mayer and Wehner (1) did their original R. F. sputtering research, it is interesting to examine the first R. F. sputtering chamber arrangement. This arrangement is depicted in Figure 2.

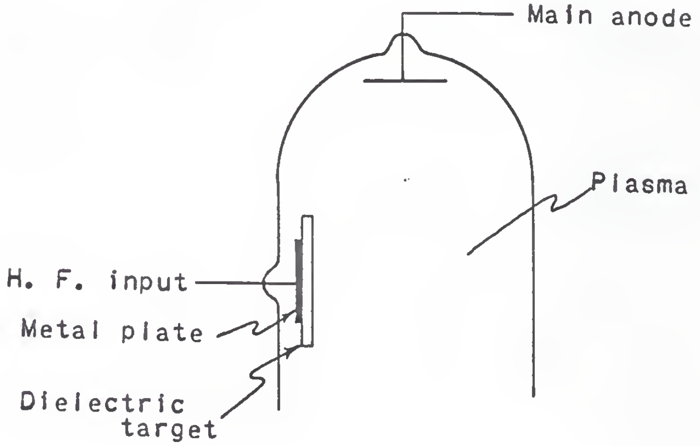

Figure 2. Original R. F. sputtering chamber arrangement.

A recently designed sputtering chamber is illustrated in Figure 3. Most of the major parts and features of this chamber will be found in any R. F. sputtering chamber now in use. In addition, if high current densities are used, water cooling is provided for the target and substrate holder.

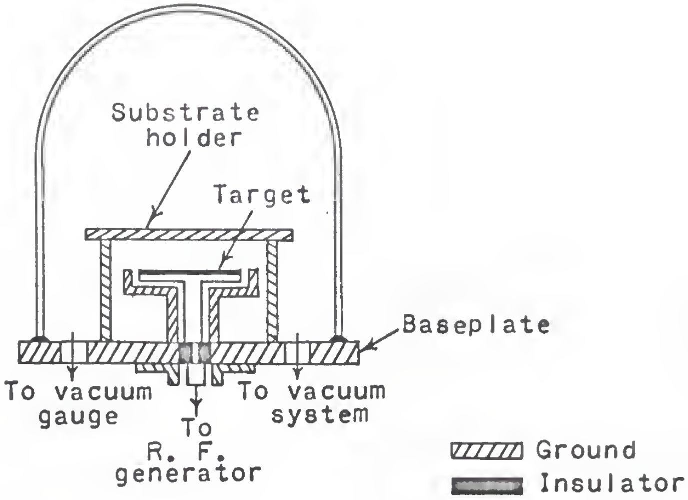

Figure 3. Typical R. F. sputtering chamber.

Considerable care must be taken in the design and construction of the sputtering apparatus. Sharp corners and rectangular geometries must be avoided in order to eliminate field concentrations. Much higher power levels are needed with a rectangular electrode than with a circular electrode to obtain the same deposition rate (3). Materials to be used in apparatus fabrication must be chosen with care. For example, teflon can be used as an insulating spacer for low power applications, but the toxic fumes given off when teflon melts limit its usefulness.

A ground shield is positioned behind the target electrode, regardless of the type of sputtering employed. The shield eliminates the sputtering of target material in undesired directions, and also prevents the dissipation of power from the back of the electrode, thus increasing efficiency.

For R. F. sputtering, the distance between the ground shield and the R. F. electrode is critical, and should be kept as close to the thickness of the cathode dark space as possible without exceeding it. (If the distance exceeds the dark space, a discharge will be established between the electrode and the shield, resulting in sputtering of the back of the electrode.)

The thickness of the dark space is proportional to the electron mean free path, and therefore inversely proportional to pressure. (At 0.02 Torr, the dark space thickness is approximately 0.8 cm.)

If the distance between the electrode and the shield is much less than the dark space thickness, the resulting low impedance at radio frequencies will mean that most of the R. F. power will be lost directly to the ground. The result will be very low sputtering rates. Best results were obtained by Davidse and Maissel (3) with spacings of 0.50 to 0.65 cm.

The separation of the target and the substrates must, of course, be greater than the thickness of the dark space. Typically, target-substrate separations of 3-10 cm are used.

The impedance of the target must be kept as low as possible so that power losses are minimized. Therefore, particularly in the case of dielectrics, the target must be kept as thin as possible. In addition, the back surface of dielectric targets should be metalized, and the targets soldered, if possible, to the support plate. Targets, however, must be kept sufficiently thick so that they do not physically warp due to the heating produced by the ion bombardment. (In the Kansas State University Solid State

Laboratory, a 0.015 inch thick molybdenum electrode was found to be unsatisfactory due to heat-induced warpage.)

For D. C. sputtering, the generator, for general purposes, should be capable of an output of 0-5 kV at a current density of two milliamps per square centimeter of target area. For a four-inch round electrode, then, the generator should be capable of maintaining an output current of about 150 ma.

For R. F. sputtering, an R. F. generator may be selected only after the frequency of operation is determined. The frequency is usually in the range of 1-20 MHz, for reasons which will be discussed later.

The generator must be able to sustain a power output of at least one to three ma/cm^2 of target area in addition to any losses in the system. Also, an impedance matching network should be included so that the system can be tuned to resonance, allowing maximum power transfer to the discharge.

If the coaxial line from the R. F. generator to the sputtering chamber is more than a few feet long, it is good practice to keep it a multiple of a half-wavelength. This is because the input impedance of a half-wavelength section is always equal to the output impedance, regardless of the standing wave ratio (SWR) value.

The most effective matching network which can be simply operated, according to McDowell (13), is illustrated in Figure 4. The operating procedure is first to tune L for maximum R. F. voltage and then tune C for maximum forward power and minimum reflected power.

An equivalent circuit of an R. F. sputtering system is depicted in Figure 5. The series capacitance C_s is included whenever

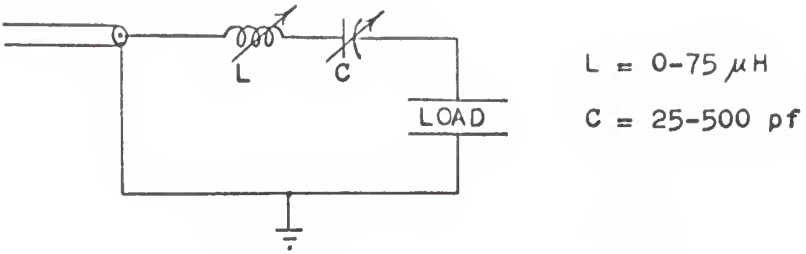

Figure 4. R. F. impedance matching network.

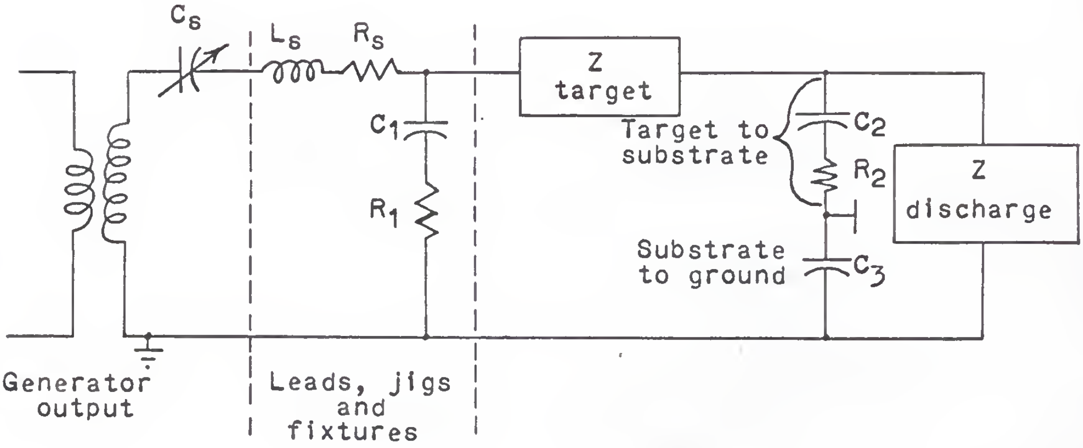

Figure 5. Equivalent circuit of an R. F. sputtering system. (After Vossen and O'Neill (21))

metals are sputtered. It may be omitted when sputtering dielectrics.

The gas most commonly used for sputtering is argon. There are three primary reasons for its use. First, it is inert and therefore does not react with the target material. Second, it has a relatively high atomic weight. As will be shown later, this has a favorable effect on the deposition rates. Third, it is available in highly pure form at a fairly low cost.

Vacuum gauging is a troublesome area in sputtering. It is definitely more difficult to do accurately than it may seem.

One problem arises from the range over which sputtering is carried out. R. F. sputtering may be done at a pressure as low as one millitorr, while high pressure or reactive sputtering is often done at pressures in excess of 100 millitorrs. Cold cathode gauges generally do not measure pressures in excess of 25 millitorrs, while thermocouple gauges generally do not measure pressures less than 10 millitorrs. The result, all too often, is that two complete sets of gauging do not agree even approximately in their common range.

The obvious answer is to calibrate the gauges against, for example, a McLeod gauge. A set of correction curves may then be used to convert gauge readings into true readings. Care must be taken to correct the true readings for whatever gas is being used in the sputtering chamber, for example, argon. This is because most gauges are calibrated for air.

To make matters worse, the vacuum reading obtained will obviously depend very much on how the gauge is positioned relative to the vacuum pump and the glow discharge. The only practical answer to this problem is to keep the gauge(s) in a fixed location, and state the location whenever publishing experimental results. The study of the variation of pressure within a sputtering chamber is an entire area in itself.

CHAPTER III

AN ECONOMICAL SYSTEM

The purchase of a sputtering system is a major investment, even for a large company or a university. Prices usually start around $6,000 for a system or $1,500 for a sputtering chamber alone. Added to this is the cost of liquid nitrogen, inert gas, target materials, substrates, cleaning equipment, et cetera. The cost of a sputtering system is thus prohibitive for many potential users, particularly universities who need this state-of-the-art equipment to provide instruction in thin film deposition.

A sputtering chamber which can be built for $100-150 and a few hours of sheet metal work is described in this chapter. This chamber is suitable for D. C. and R. F. sputtering, and can be used at power levels up to about 250 watts. The chamber has been constructed and tested in the Kansas State University Solid State Laboratory, and has been found to be completely adequate for instructional purposes and basic research. See Figure 6 for a sectional illustration of the chamber.

The housing for the chamber is a 12 inch length of 6 inch diameter pyrex glass pipe. A teflon gasket is placed on each end of the pipe to provide a vacuum seal to the base plates. The base plates are one inch thick aluminum plates which have been machined to a diameter of 10 inches.

The upper plate is tapped in the center for a $\frac{1}{4}$ inch Swagelock

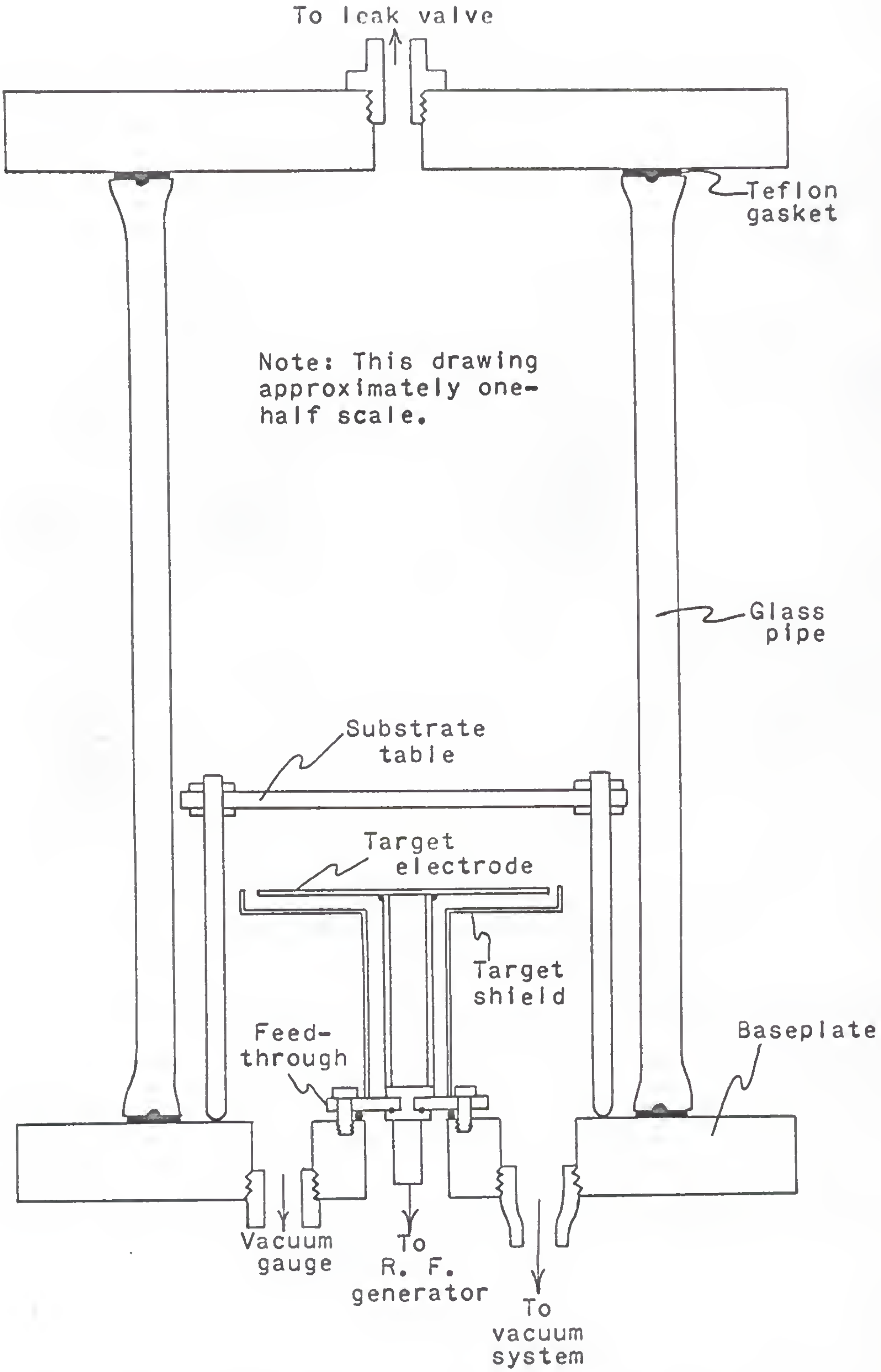

Figure 6. Sectional view of economical sputtering chamber.

fitting. A ¼ inch copper tube connects this fitting to the leak valve, a Whitey 21RS4.

The lower plate is drilled in the center for the ceramic feedthrough. It is tapped off-center for the thermocouple vacuum gauge and also for the pumpdown opening to the vacuum system. (In the laboratory model, a cold cathode gauge is also mounted off-center.) The thermocouple gauge is a Virtis Model 10-324.

The ceramic feedthrough is an old microwave fitting found in a box of spare parts. The copper target electrode is a four inch diameter 1/10 inch thick copper disc which was silver soldered to a 5/8 inch diameter copper tube. The target shield was fabricated from sheet aluminum and a steel tube and was assembled with the aid of a pop-riveting tool. The substrate table was made from 3/16 inch thick aluminum, and is supported by four 1/4 inch diameter aluminum rods.

Except for the leak valve, pyrex pipe and teflon gaskets, all of the material used in the chamber was scrap material obtained from various departmental shops at Kansas State University. (The one inch thick aluminum had been purchased as surplus material from the State of Kansas.) The leak valve cost $35, the pyrex pipe cost $40, and the two teflon gaskets cost $9. Making allowance for the cost of machining and tapping the base plates, their cost was approximately $16, resulting in a total cost for the chamber of approximately $100. The necessity of purchasing the sheet metal, ceramic feedthrough, "O" rings, et cetera, would not have increased the total cost of the chamber beyond $150.

Figure 7 is a photograph of the completed sputtering chamber.

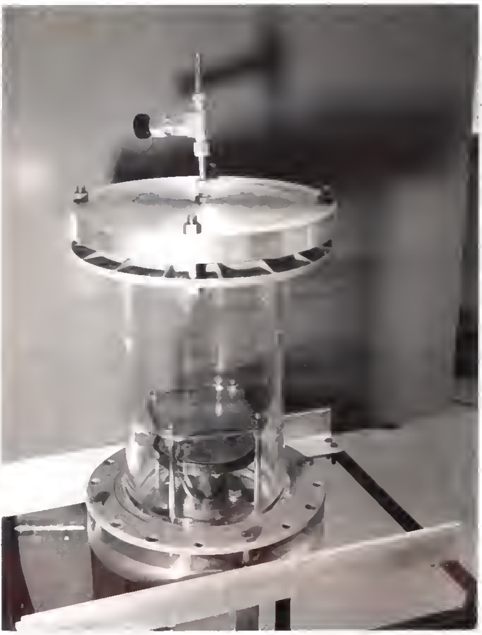

Figure 7. Completed sputtering chamber.

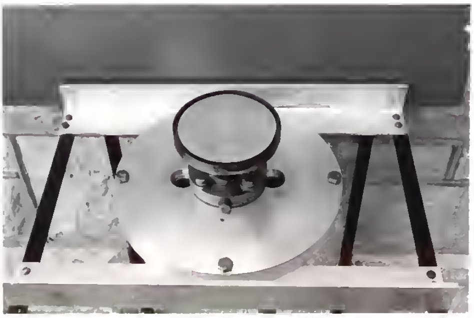

Figure 8. Lower base plate of sputtering chamber.

Note that cast iron flanges are being used to clamp the base plates in position. These flanges add stability, but are not absolutely necessary. Figure 8 is a photograph of the lower base plate with the target electrode and target shield attached. In Figure 8, the hole immediately to the left of the feedthrough marks the location of the cold cathode gauge, the hole immediately to the right of the feedthrough is for vacuum pumpdown, and the small hole in front of the feedthrough is the location of the thermocouple gauge.

An economical sputtering system was assembled by minimizing the cost of the equipment used with this sputtering chamber. The vacuum pump used was a $250 Welch Duo-Seal mechanical pump. The thermocouple gauge cost $87. A 200 watt amateur radio transmitter can be fabricated for $200. (In the laboratory, a Westinghouse 10 MHz industrial R. F. generator was utilized.) Eliminating the cold trap, which was found to be unnecessary for instructional purposes, and allowing $150 for the sputtering chamber, the resulting total system price is approximately $700.

Although lacking the sophistication of multi-thousand dollar systems, the $700 system performs quite satisfactorily. The only system failure in the laboratory occurred while coupling 450 watts into the sputtering chamber. Excessive heat caused the collapse of the ceramic feedthrough. For data obtained with this system, refer to the experimental results chapter.

CHAPTER IV

DEPOSITION RATES

Perhaps the most common area of sputtering research has been that connected with deposition rates, and how they are influenced by various factors. In general, the deposition rate increases very rapidly in the low ion energy (less than 10 keV) region and levels out in the medium ion energy (10-50 keV) region. The rate then decreases in the very high energy range, due to deep penetration of the ions, which minimizes energy ultimately transferred to the surface. Other variables which affect deposition rate are the target material, sputtering gas, pressure, electrical parameters of the discharge, magnetic fields which are present, target area, target-substrate separation and angle of ion incidence.

The sputtering yield, or number of atoms ejected per incident ion, is dependent upon the angle of incidence, which is measured with respect to the normal to the target surface. As the angle of incidence is increased, the ion spends more of its energy in collisions near the surface, and a higher yield results. This is illustrated in Figure 9. The quantitative values for the yield vary, of course, with such parameters as ion energy, system pressure and materials used, but the increase in yield with angle of incidence is valid in all cases for which data are available (12). This angular yield dependence suggests that, at least in certain low energy ranges, the sputtering process may not be described solely

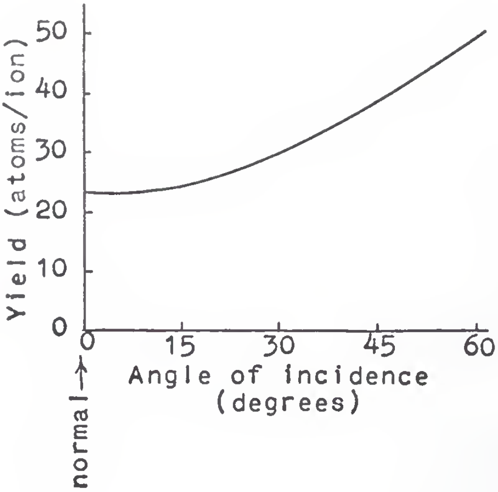

Figure 9. Variation in yield with angle of incidence.

by a non-directional transfer of energy, but also by a transfer of momentum from the ions to the target atoms as in hard sphere collisions.

As seen in Figure 9, sputtering yields can be quite large. For instance, 50 keV xenon ions incident normally on copper eject 20 atoms per projectile. In a lower energy range, 5 keV argon ions incident on copper eject an average of five atoms per projectile.

Elastic constants of the target and the strength of the atomic or molecular bonds are also important parameters. In general, insulators can be divided into three groups. First are multicompound crystalline solids, which have the lowest deposition rates. Second are simple crystalline solids. Third are non-crystalline solids (glasses), which have the highest deposition rates.

Sputtering frequency has little influence on the deposition rate, as long as it is in the low mHz range. Several experiments carried out between 2 and 27 mHz demonstrated that the frequency had little effect on the deposition rate in this range (4). A frequency of 13.56 MHz is frequently used because of Federal Communications Commission licensing regulations.

R. F. sputtering is commonly done with a chamber pressure of about 5-50 millitorrs. The variation of rate within this range is usually not great, but different researchers have reported varying results. Vossen and O'Neill (21) reported that highest deposition rates occur at pressures of about 2-15 millitorrs, and that the rate is fairly constant over this range. Cash, Cunningham and Keene (2) found that deposition rates decreased appreciably as the pressure was increased above five millitorrs, probably due to increased scattering of the sputtered target particles. They also reported that the rate remained fairly constant down to approximately one millitorr. Obviously, when the pressure drops low enough that the sheath width approaches the target-substrate separation, the deposition rate will drop to practically zero.

Deposition rate is inversely proportional to the target-substrate separation distance. This is logical, and means only that the substrates should be kept within approximately 10 cm of the target.

Deposition rate as a function of target area has been found to follow the 3/2 power law (21). This is because the impedance of the target is inversely proportional to the target area. (Therefore the discharge power is proportional to the target area.) This is also because of the hemispherical distribution of the ejected material.

For both D. C. and R. F. sputtering, the deposition rate has been found to be directly proportional to the discharge power. This is illustrated in Figure 10 for a particular R. F. system configuration (21). The data for Figure 10 were obtained in the presence of an axial magnetic field of 70 gauss.

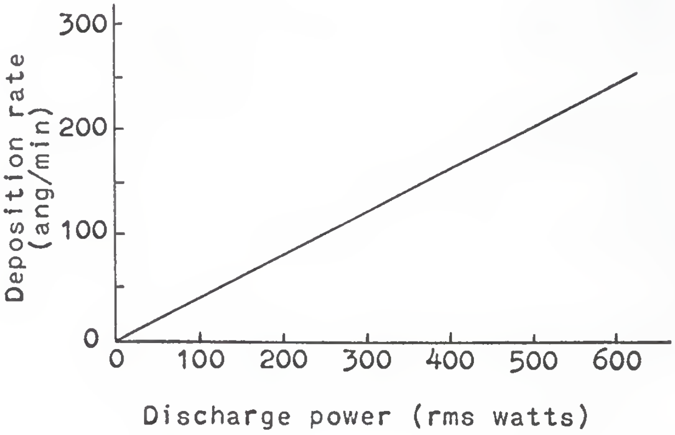

Figure 10. Deposition rate vs. discharge power.

The ratio of the incident ion mass to the mass of the target atom affects the deposition rate. Very roughly, sputtering is expected to be most prolific if the incident ion mass is equal to that of a target atom (15). This means that, for maximum deposition rates, the material to be sputtered determines the choice of inert gas.

The temperature of the substrate significantly affects the

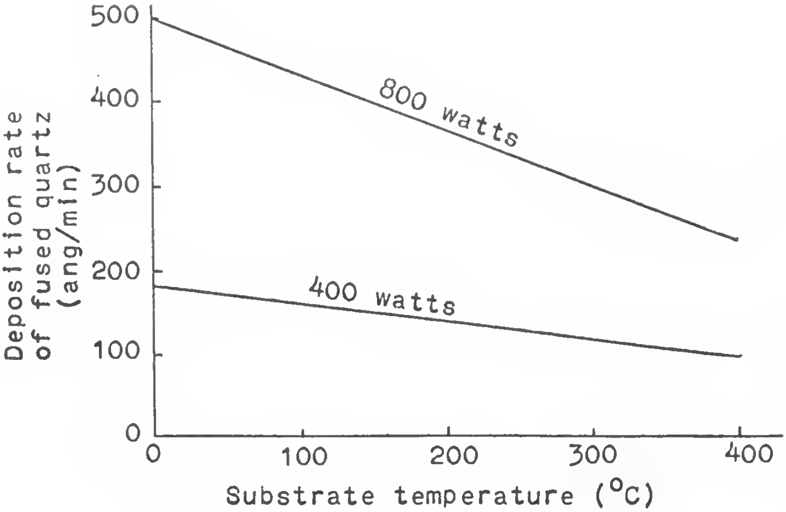

Figure 11. Deposition rate vs. substrate temperature. (After Davidse (4))

deposition rate, as is illustrated by Figure 11. It is therefore obvious that a uniform substrate temperature is necessary if a uniform film thickness is to be obtained.

The quality of the contact between the substrate and ground (the substrate holder) has a marked effect on deposition rate. If C_1 is defined as the substrate to ground capacitance and C_2 is defined as the target to substrate capacitance, assumed constant, then it follows that C_2/C_1 is a measure of the quality of the ground. Typical results obtained by Vossen and O'Neill (21) by isolating the substrate from ground by varying amounts are illustrated in Figure 12.

Of considerable interest in the study of R. F. deposition rates is the effect of superimposing a magnetic field on the glow discharge. Using Helmholtz coils, the magnetic field midway

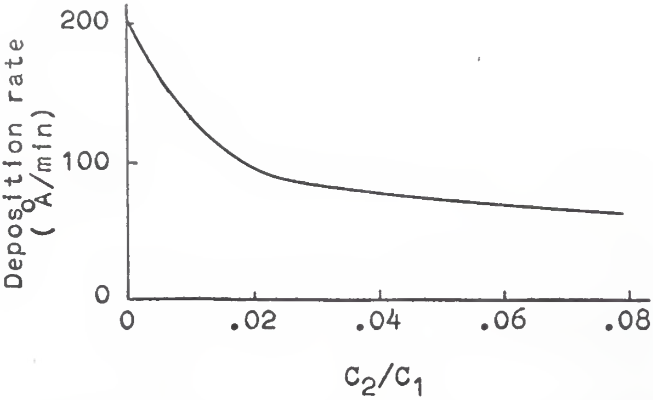

Figure 12. Deposition rate vs. quality of ground.

between the coils can be made to be very uniform over a considerable region (20). The field is directed so that it is parallel to the axis of the sputtering chamber, that is, perpendicular to the target and substrate holder.

When sputtering dielectrics, the effect of the magnetic field is to reduce the pressure at which a glow discharge can be started and maintained to about one millitorr. Also, matching the R. F. generator to the load is easier with the field present (3).

For dielectrics, a magnetic field of 100 gauss gives up to twice the zero-field deposition rate. A saturation effect takes place at higher field intensities, as is illustrated by Figure 13. (Also, it should be noted that saturation occurs at higher field intensities for higher R. F. power levels.)

The reason for the increased deposition rate is that the magnetic field constrains electrons to follow helical paths, the radii of which are:

$$r = \frac{m_e v \sin\phi}{eB},$$ where v = electron velocity,

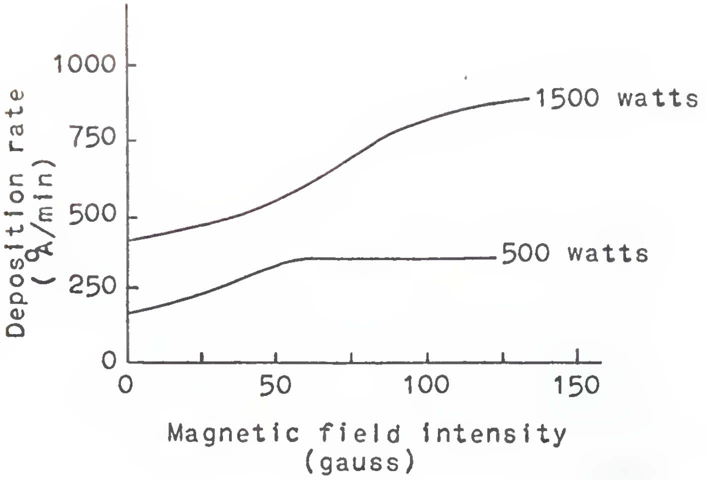

Figure 13. Deposition rate vs. magnetic field intensity. (After Davidse (4))

ϕ = electron emission angle, m_e = electron mass, e = electronic charge and B = magnetic flux density. The net effect, then, is to lengthen the electron path and increase the probability of a collision, thus yielding higher ion density.

The largest increase in deposition rate and uniformity of film thickness occurs when the coil and substrate are concentric. The magnetic field has been found to greatly improve the uniformity of film thickness over large substrate areas, probably by scattering the emitted species slightly (21).

It is interesting to note that although the magnetic field increases deposition rate for dielectrics, experiments involving the R. F. sputtering of metals indicate no increase in deposition rate for metal sputtering. Variations in the magnetic field from 0-200 gauss did not cause any change in film thickness (2).

CHAPTER V

OTHER SPUTTERING PHENOMENA

Self-Bias Phenomenon

One of the most important phenomena associated with R. F. sputtering is that of self-biasing. The surface potential of the target becomes negative with respect to the potential of the plasma. This negative self-bias, V_b, is illustrated in Figure 14.

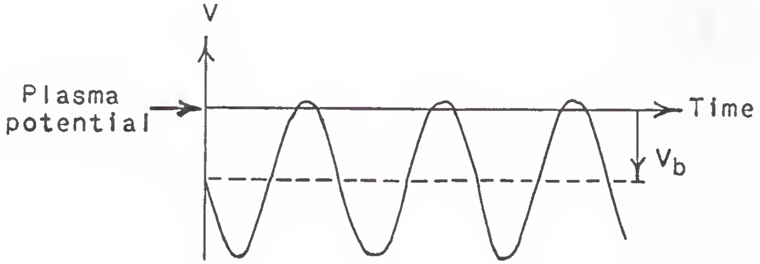

Figure 14. Potential of target surface, V, vs. time.

The necessity for negative self-bias may be explained by examining particle mobility. Since the mobility of a particle is proportional to its charge and inversely proportional to its mass, electron mobility is approximately 10^5 greater than the mobility of argon ions. Therefore, more electrons collect on the cathode during each cycle than argon ions. Because of the capacitive nature of a dielectric target, and the blocking capacitor inserted when sputtering metals, electrons accumulate on the cathode, resulting in the negative self-bias V_b.

This self-bias phenomenon places certain constraints upon the sputtering frequency. If the sputtering frequency is too low, no D. C. potential will build up on the target because enough ions will strike the target surface during the negative half of each cycle to neutralize the negative surface charge. Experiments have shown that the frequency must be at least on the order of 10 KHz for any ion sheath to form (4). Although frequencies in the low megahertz range are usually used, experiments in the microwave range have been conducted.

Difficulties encountered with the neutralization and accumulation of charge would, of course, disappear if beams of neutral atoms were utilized rather than ions. It is difficult, however, to produce neutral beams with high flux densities in the energy range of interest.

Consider the effect of positive charge buildup on the target surface of a dielectric target. Assume, for calculation purposes, that ion current (I_i) is independent of time. Then the potential of the dielectric surface, during the negative half cycle, will decrease in absolute value at a rate given by $dV/dt = I_i/C$, where C = capacitance of the dielectric target and I_i = ion current. Considering a current of 1 ma/cm^2 and a capacitance C of 20 pf/cm^2, $dV/dt = 5 \times 10^7$ volts per second. Therefore, if the decrease in the potential of the dielectric surface due to positive charge accumulation is about 100 volts per cycle, the frequency must be on the order of 500 KHz.

High Pressure Phenomena

Utilizing relatively high chamber pressures is one method of controlling sputtering growth processes. For example, the deposition of tantalum films at relatively low voltage (1-3kV) and high argon pressure (20-120 mT) has been found to result in films of 1000 ohms per square for a 200 angstrom film (14). These films were also found to have essentially zero temperature coefficients.

The films consisted primarily of tantalum with very small crystallite sizes in a form which had not been previously observed. This form of tantalum was later named beta tantalum. Because of the small crystallite size, the electron mean-free path was quite small and the temperature coefficient was relatively independent of the thermal lattice vibrations.

Schuetze, Ehlbeck and Doerbeck (16) reported similar results. They also observed that since the partial pressures of the gaseous impurities in the sputtering chamber were lower than 10^{-5} torr, the observed density of the sputtered films could not be explained by impurity content. They accounted for the higher resistivity in a low-voltage sputtered film by stating that the film is composed of loosely packed atoms.

They found that the resistivity of the sputtered film varied inversely with the sputtering voltage, as illustrated in Figure 15. (A constant value of sputtering current was maintained by varying the flow of inert gas into the chamber.) Almost no influence of current or sputtering time on resistivity was found, however. As a typical result, a 500 angstrom tantalum film sputtered at 1500 volts was found to have a resistivity of 4000

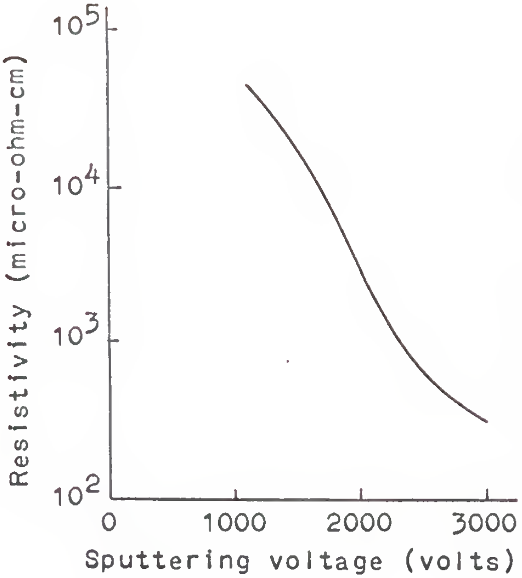

Figure 15. Resistivity of tantalum sputtered films versus sputtering voltage. (After Schuetze, Ehlbeck and Doerbeck (16))

ohms per square.

In order to obtain stable values of resistance, the films were aged for 25 hours in 200°C air. The temperature coefficient of resistance (TCR) was then measured. A TCR of zero was measured for films deposited at 2.25 kV. This result was found to be reproducible within 80 ppm/°C. (Films sputtered at less than 2.25 kV had negative TCR's, while those sputtered at more than 2.25 kV had positive TCR's.)

The environmental effect of the inert gas is very important in the sputtering process, and becomes a dominant factor in high pressure sputtering. In an experiment conducted by Wolsky (25), germanium atoms were being deposited at the rate of 10^{15} atoms/ cm^2·min. At the same time, the substrate surface was being bombarded with approximately 10^{19} argon atoms/cm^2•min. Wolsky concluded that the argon atoms reduce the effective temperature of the surface atoms.

Some research in high pressure sputtering has been done in the Kansas State University Solid State Laboratory. It is presented in the chapter on experimental results.

Contamination Phenomena

Of considerable importance are the factors which give rise to the contamination of a sputtering system, and the effects which contamination has on the system. Of particular interest, of course, are the target and substrate surfaces.

In some cases, the electrodes themselves can act as catalysts for contaminating reactions. For example, the yield of ozone is enhanced in a glow discharge when the electrodes are made of aluminum (10).

Several authors have shown that the gas content of the target may be several orders of magnitude greater than the normal solubility of the gas. The result is that drastic changes occur in the physical properties of the target material. This was noted particularly for targets with thin oxide layers (10).

The contamination of a target may then, in some cases, display a regenerative property. That is, a target may acquire a thin oxide layer, giving rise to an abnormally high gas content which in turn gives rise to further contamination, perhaps in the form of reactive products.

The rate of contamination can be greatly affected by current density and the energy of the incident ions. This is because these parameters, among others, determine the rate at which undesired reaction products are removed from the cathode.

Ionic bombardment of a target has been found to affect the oxidation rate of the target surface. It has been shown that, at least for some target materials, bombarding the target with 1 keV ions increases the oxidation rate of the target surface by more than a hundred times (10).

One method used to rid the target of initial contamination is a high temperature bake-out with subsequent prolonged pumping to a pressure of 10^{-9} torr. This method, however, is not practical for most sputtering applications. More often, the method used is ion bombardment with an inert gas. In other words, sputtering is carried out for a few minutes onto a shutter mechanism which is placed in front of the substrates.

To avoid oxide formation on the target during sputtering, the ion energy must be such that the rate of ejection of particles is greater than the rate of oxidation of the target. Some undesirable reactions on the target surface can be avoided by water cooling the target. The substrate holder is also often water cooled. The cooling not only reduces undesirable chemical reactions, but also maintains the electrodes at a constant temperature, enabling a reproducibility of results not otherwise obtainable.

As would be expected, contamination of the substrate is an ever present problem. Kay (10) found that the problem of film growth with respect to the residual gaseous species arriving at the substrate is essentially that of physical adsorption. He defined physical adsorption as those interactions arising from van der Waal's forces between the incident atom and the substrate lattice. These forces are similar in magnitude to the heat of sublimation of the rare gases.

When considering inert gas contamination, it is important to examine the rate of arrival of cathode-ejected material compared with the rate of arrival of inert gas atoms. This is determined both by the chamber pressure and by the energy of the ions incident on the target, which determines ejection rate at the target.

Of the various methods used to reduce substrate contamination, one of the most recent and most successful is substrate bias. Substrate bias during D. C. sputtering, for example, has been found to effectively reduce oxygen contamination in nichrome films (19). It appears that the resputtering of adsorbed impurity atoms, in particular oxygen, is responsible.

Data obtained by Stern and Light (19) at a bias of -150 volts (cathode potential of -2.0 kV) indicate that the oxygen content of films sputtered in a high partial pressure of oxygen (10^{-5} torr) does not differ appreciably from those sputtered in a pure argon atmosphere where the partial pressure of oxygen was approximately 2×10^{-7} torr. Furthermore, it appears that substrate bias assists in the orientation of the film.

Several types of substrate bias are presently in use. These include D. C. bias while D. C. sputtering (19), D. C. bias while R. F. sputtering (13), and R. F. bias while D. C. sputtering (22). Not only does the biasing technique reduce substrate contamination, but it also increases deposition rates.

CHAPTER VI

EXPERIMENTAL RESULTS

System Description

The sputtering chamber used for this research was described earlier in Chapter III. Using a Welch 1405 Duo-Seal vacuum pump, an ultimate chamber vacuum of 3×10^{-4} torr was attainable. Vacuum measurements were made with a CVC Model GPH-100A cold cathode gauge and a Virtis Model 10-324 thermocouple gauge. A liquid nitrogen trap was used for some experiments, but was omitted to promote system simplicity for data presented in this chapter.

The power supply used for D. C. sputtering was a CVC Model LC-031 capable of a constant output of 350 ma at 5000 volts. The R. F. power supply was a 10 MHz Westinghouse industrial R. F. generator with a rated input to the final power tube of 1000 watts. For R. F. sputtering, the generator was coupled with RG-8/U coaxial cable directly to the sputtering chamber. A 500 pf capacitor was inserted at the base of the sputtering chamber when sputtering conductors.

The system is illustrated in Figure 16. At the left of the sputtering chamber is the D. C. power supply. The cold cathode gauge is on top of the supply. To the right of the sputtering chamber is the R. F. generator. The readout for the thermocouple gauge is resting on the left end of the generator. The vacuum pump is located beneath the sputtering chamber.

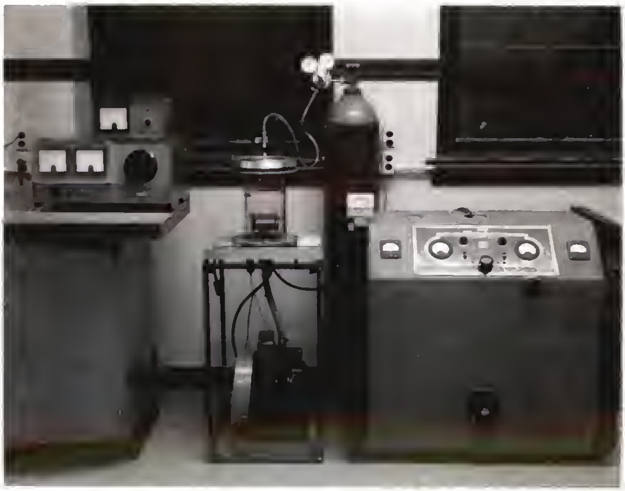

Figure 16. Sputtering system.

Tuning the R. F. Discharge

The usual method for tuning an R. F. sputtering system is to adjust the R. F. generator and matching network for maximum R. F. power coupled to the load; i.e., minimum standing wave ratio. Another method is suggested by the properties of a glow discharge.

A discharge is maintained by electrons produced at the cathode as a result of positive ion bombardment. Each electron must produce enough positive ions to release a new electron at the cathode. Although it is generally agreed that the negative glow is a result of excited atoms returning to the ground state, there is disagreement as to the cause of the cathode glow.

Kay (10) states that when the positive ions are neutralized, the decay of their excitation energy gives rise to the cathode glow.

Wehner (24) believes that sputtered copper ions with an average lifetime of 10^{-8} seconds cause the cathode glow when they are de-excited.

Whatever the cause of the glow, it provides a good indication of the sputtering rate. Tuning the discharge for maximum cathode glow is equivalent to tuning for a maximum density of sputtered particles. Thus an excellent method of tuning the R. F. discharge is available which does not require the use of forward and reflected R. F. power meters, et cetera. This method has been tested in the laboratory and found to be a sensitive tuning technique.

Substrate Stress

The effects of stress were observed when microscope slide substrates were being held in place by stainless steel clips, as illustrated in Figure 17. The clips held the slides up against the bottom of the substrate table (see Figure 6).

One of the machine screws was accidentally overtightened. The resulting copper film had an area of non-adhesion along the edge of the clip in the vicinity of the overtightened screw, as illustrated in Figure 18. The area of non-adhesion was marked by bubbles (or blisters) of copper film.

The conditions were repeated for various patterns of substrate stress, with the same result. The film along the edge of the clip near an overtightened screw always had an area of non-adhesion.

Authors have variously reported both tensive and compressive stress in sputtered films. The copper bubbles or blisters which

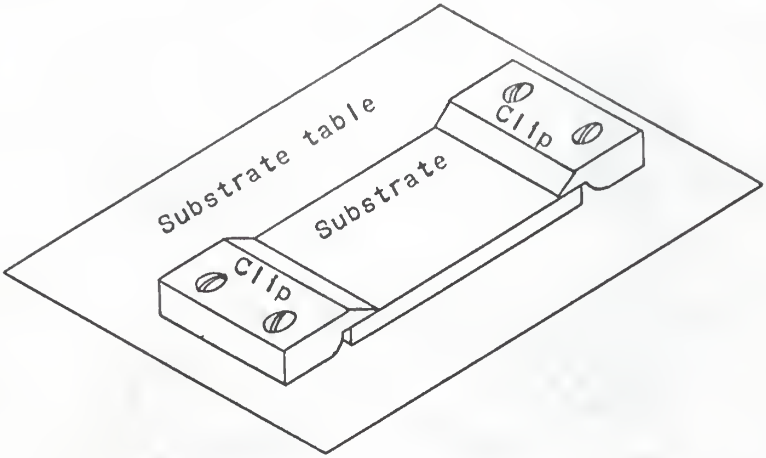

Figure 17. Substrate clamping arrangement.

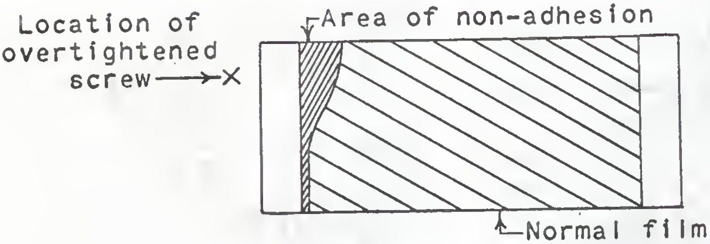

Figure 18. Area of film non-adhesion due to substrate stress.

were observed, however, clearly demonstrated the presence of compressive stress in the films of this experiment.

The films were otherwise of high quality and high conductivity. They were D. C. sputtered at 1 ma/cm^2, 2000 volts and 10×10^{-3} torr. The overtightening of the screws apparently created dislocations or other surface imperfections in the glass substrates which caused the non-adhesion to occur.

High Pressure Effects

Unusual results were obtained when sputtering copper at relatively high chamber pressures. For example, for a chamber pressure of about 60×10^{-3} torr and a current density of 0.5 ma/cm^2, a 1200 angstrom film was obtained which had a resistivity of 1700 ohms/square. (The sputtering rate was 120 angstroms per minute.) The film was uniform and black in color. Qualitatively similar results were obtained with molybdenum.

Results obtained by Wehner (24) with a scanning electron microscope indicate that the films are composed of very loosely packed atoms of copper. Apparently, aggregates of copper atoms are formed before reaching the substrate, and these particle clusters are deposited on the substrate, rather than single atoms. If the partial pressure of oxygen is sufficient, oxygen atoms attach themselves to these particle aggregates. The film then consists of a sort of modified oxide, again very loosely packed. These results are roughly analagous to the results obtained with beta tantalum.

To aid in the understanding of phenomena which occur at high chamber pressures, a review of kinetic gas theory is helpful. It has been well established by several researchers that the sputtered atom is ejected from the target surface with an average energy of 3-10 electron volts. (Larger energy levels have been observed for oblique ejection directions.)

This means, for molybdenum, that the velocity of the ejected particles ranges from 2.5×10^5 cm/sec for 3 eV particles to

4×10^5 cm/sec for 10 eV particles. The average velocity v_a of argon atoms striking the substrate is $14{,}551\sqrt{\frac{T}{M}}$ cm/sec, where M is the atomic mass and T is the temperature of the argon gas adjacent to the substrate in degrees Kelvin (5). Argon atoms are therefore bombarding the substrate with an average velocity of 6×10^4 cm/sec, assuming a substrate temperature of 300°C.

Assuming a deposition rate for molybdenum of 100 angstroms per minute, the substrate is being coated with approximately 10^{14} atoms/cm^{2}sec. The number of molecules of a gas that strike a unit area in unit time is given by 3.513×10^{22} $P/\sqrt{MT}$ cm^{-2}sec^{-1} (5), where P is the gas pressure in mm Hg. Assuming a chamber pressure of 50×10^{-3} torr and a gas temperature adjacent to the substrate of 300°C, 10^{19} atoms of argon will strike each square centimeter of target area every second. There are therefore approximately 10^5 argon atoms striking the substrate for every atom of molybdenum.

It thus is obvious, both from velocity magnitudes and bombardment densities, that the argon gas in the sputtering chamber plays an important role in the formation of the thin films on the substrate. One effect which has been noted is that the argon atoms reduce the effective temperature of the surface atoms of both the target and the substrate (25).

This high density of argon in the chamber almost certainly affects the formation of aggregates of atoms, perhaps through collisions en route to the substrate. Any such collision process would slow the particles to something less than their ejection velocities, perhaps accounting for the loosely packed structure on the substrate. High pressure effects will continue to be a subject of research in the laboratory.

System Impedance Characteristics

Characteristic impedance curves may be determined for a sputtering chamber much as for a vacuum tube or semiconductor device. These curves provide a means by which a particular sputtering system may be uniquely identified.

Figure 19 is a set of curves for the sputtering chamber described previously. The curves were taken with the copper electrode in place. The pressures have been corrected for argon gas, but the GPH-100A gauge on which they were taken was not calibrated against a standard.

Note that the curves are similar to those for a diode, which might be expected. The pressure was adjusted from a no discharge condition to a saturation condition. Current readings were taken on a Triplett Model 327-A milliammeter.

The ordinate of Figure 19 was plotted as current/area. This is a valid unit of comparison, regardless of the size of the system. The characteristics are thus more universal in nature.

Similar characteristics can be plotted for R. F. systems. A difference between systems in variable losses might tend to decrease the universality of R. F. characteristics, but they would still be very useful.

These characteristics, for D. C. or R. F. chambers, once plotted and calibrated for pressure, can serve as a reference guide. For instance, if, for the chamber of Figure 19, the chamber voltage is 1900 volts and the current density is 1 ma/cm^2, it

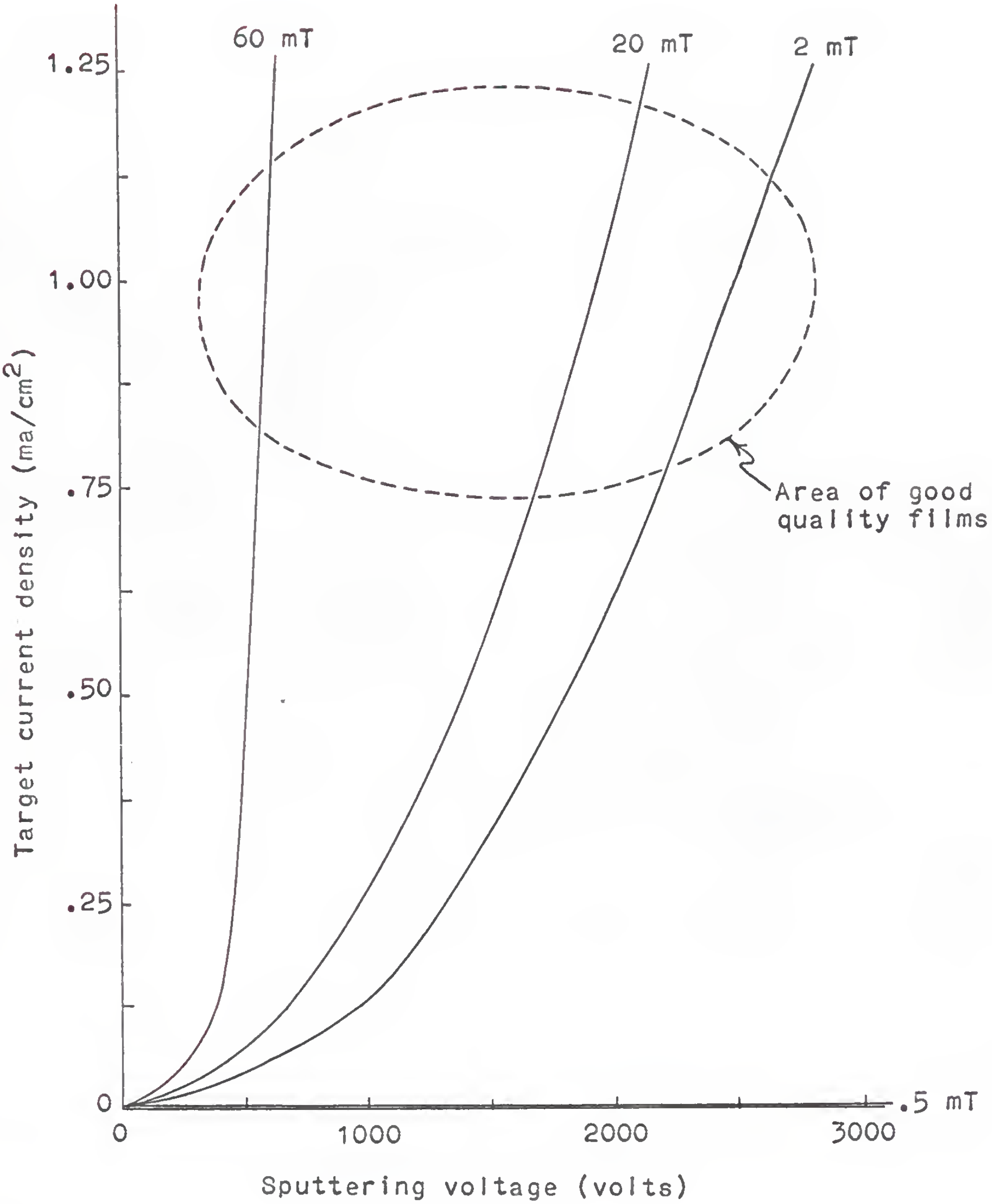

Figure 19. Impedance characteristics for D. C. chamber with copper electrode.

is immediately evident that the chamber pressure is 20 x 10^{-3} torr. Constant vacuum recording is thus made unnecessary.

Preliminary evidence indicates that an area of the characteristic curves may be outlined which will show the area in which good quality, highly conductive films will be obtained. The possible form of such an area is outlined by a dotted line in Figure 19. Further research will be conducted in the laboratory to ascertain the validity of this assumption.

Conclusion

The amount of research accomplished thus far in the laboratory has made evident the vast amount that remains to be done. Emphasis will continue to be placed on high pressure phenomena and the use of impedance curves to characterize sputtering systems.

A stupendous amount of material has been written about sputtering, but, as is illustrated by the puzzling action of R. F. sputtering a metal with a magnetic field, there is still much that is not yet understood. This lack of understanding has been, to a great degree, because of the scarcity of reliable experimental data.

Wehner, one of the originators of the R. F. sputtering technique, states that sputtering is still, to a great extent, "black magic". The sheer volume of research being done, however, will surely alleviate this situation.

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CONSIDERATIONS IN THE DESIGN AND UTILIZATION OF SPUTTERING SYSTEMS

by

RUSSELL CARLEY EBERHART

B. S., Kansas State University, 1965

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Electrical Engineering

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1969

ABSTRACT

Although sputtering phenomena were first observed more than 100 years ago, sputtering as a method of thin film deposition has only recently enjoyed a rapid increase in utilization. Recent experimental data and apparatus technology are examined.

The physical process of sputtering is reviewed and the nature of the ejected particles is discussed. The events leading to ionization in a glow discharge are enumerated. The three main types of sputtering, D. C., R. F. and reactive, are defined and reviewed. Properties and advantages of each type are discussed.

Apparatus technology is examined in some detail. The original R. F. system configuration of Anderson, Mayer and Wehner is compared with more recently designed systems. Important areas of chamber design and fabrication technique are discussed, including electrode geometry, shield spacing and target impedance. Considerations governing the choice of each component of a sputtering system are reviewed.

An economical sputtering system which can be assembled for about $700 is described. A sputtering chamber which can be built for $100-150 and a few hours of sheet metal work is described in some detail. The chamber is suitable for D. C. and R. F. sputtering and can be used at power levels up to about 250 watts.

Perhaps the most common area of sputtering research has been that of deposition rates, and how they are influenced by various

factors. The variations in deposition rate with energy level, target material, sputtering gas, chamber pressure, electrical discharge parameters, magnetic fields, target area, target-substrate separation and angle of ion incidence are examined. Other sputtering phenomena, including that of self-bias, those associated with high pressures and those related to contamination, are discussed.

It has been found that the cathode glow in an R. F. sputtering system provides a good indication of the sputtering rate. The system can therefore be tuned for maximum deposition rate by reference to this glow.

The effects of substrate stress have been observed. The areas of non-adhesion in the vicinity of stress points indicated that compressive stress was present in the experimental films.

High resistivity films were obtained when sputtering at relatively high chamber pressures. Although dark in color, indications are that they are composed of very loosely packed atoms of target material.

It has been found that characteristic impedance curves may be determined for a sputtering chamber much as for a vacuum tube or semiconductor device. Preliminary evidence indicates that an area of the characteristic curves may be outlined which will show the area in which good quality, highly conductive films will be obtained.